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INFLUENCE ACCUMULATIVE PLASTIC DEFORMATION ON STRUCTURE  
AND PROPERTIES OF MG – ZR ALLOY

VLIV VÍCENÁSOBNÉ PLASTICKÉ DEFORMACE NA STRUKTURU A VLASTNOSTI  
SLITINY MG – ZR

**Abstract**

One of the boisterously developing areas is the development of nano-structural materials, which at present belongs to the priority areas of scientific research aimed at materials and also at forming technologies all over the world. This concerns specifically forming of non-ferrous metals and their alloys. Significance of use of these materials grows especially in automotive industry, in military and space industries.

This paper informs namely on ECAP technology investigations that have been oriented by overall objectives of acquiring new knowledge concerning deformation resistances, stress condition impacts and structure and properties of nonferrous metals and its alloys. Magnesium - zirconium alloy for investigation was used.

**Abstrakt**

Jednou z intenzívně se rozvíjejících oblastí je vývoj nanostrukturálních materiálů, který patří v současné době k prioritním oblastem vědeckého výzkumu v oblasti materiálů i tvářecích technologií ve světě. Jedná se zejména o tváření neželezných kovů a jejich slitin. Roste význam jejich použití zejména v automobilovém průmyslu, ve vojenském a kosmickém průmyslu.

Príspevok sa zaoberá zejména aplikací technologie ECAP, zaměřenou na rozšíření znalostí týkajících se hodnocení deformačních odporů, působících napětí a jejich vlivu na strukturu a vlastnosti studovaného materiálu. Ke studiu byla použita slitina hořčíku a zirkonia.

**1 INTRODUCTION**

All detailed investigation of materials becomes more and more important aspect due to growing consumption of materials and ever-increasing requirements to them.

In order to be able to achieve safe and reliable operation of these heavily exploited structures and at the same to reduce permanently their mass and bring thus economies of materials, it is necessary not only to use new materials with enhanced properties, but also to utilise better the properties of the existing materials. This requires in-depth knowledge of material (structure) characteristics, as well as their straining, which corresponds to the given exploitation conditions of the structure. These issues are closely related also to the implementation of new technologies of their production and subsequent treatment and processing, be it new technologies of heat treatment, forming and others.

One of the intensive advancing areas is the development of nano-structural materials, which at present belongs to the priority areas of scientific research aimed at materials and also at forming technologies all over the world. This concerns specifically forming of non-ferrous metals and their alloys. Non-ferrous metals and their alloys can be recycled very well and they replace more and more the steels. At the same time costs of production of products made of these materials are substantially

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falling down. Significance of use of these materials grows especially in automotive industry, in military and space industries. The leading world car manufacturers, such as Opel, Audi, Jaguar, Ford, Fiat, Volvo and Toyota at present began the development of a completely new concept of a small vehicle with high portion of aluminium and its alloys. Aluminium alloys with ultrafine grained structure are used as basic original material. Development of its structure is achieved by techniques using for obtaining of nano-structural materials. Obtaining of ultrafine grained structure in initial material leads to substantial increase in plasticity and it enables forming of materials in conditions of “super-plastic state” [1-6]. Achievement of the required structure depends primarily of the geometry of the tool, number of passes through the die, obtained magnitude of deformation and strain rate, temperature of the process and lubrication conditions.

In many technical processes of forming the deformation is substantially greater than at the tensile test. Upsetting or torsion tests have already been used for a long time at investigation of strengthening behaviour and development of material structure as they enable achievement of substantially higher degree of deformation than tensile tests [7]. Approximately 10 years ago this field of research experienced and unexpectedly big development and considerable expansion. These new activities demonstrated at the beginning of the nineties, that it is possible to manufacture nano-crystalline metallic materials by very high plastic deformation at low homological temperatures. It is possible to achieve on ductile metallic materials at the tensile test a deformation from 30% to 70%. At the torsion test it is possible to achieve on the same materials several hundreds percent. Obtaining of nano-crystalline structures requires typical magnitudes of deformation of the order from 100 to 1000% [1].

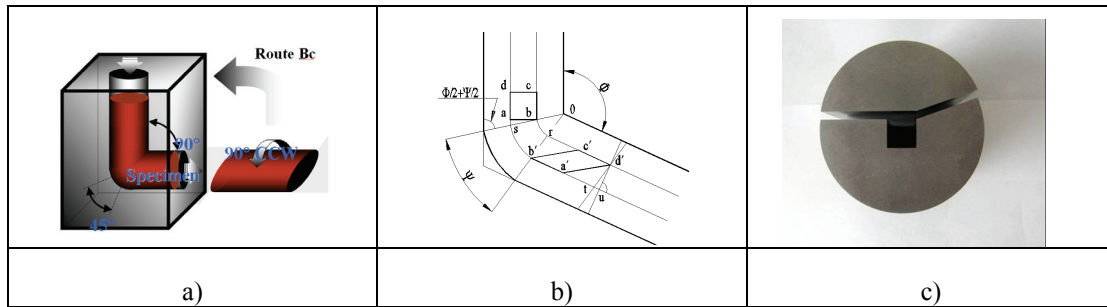
High deformation at comparatively low homological temperatures is an efficient method for manufacture of ultrafine grained massive materials. New technologies, which use high deformation for obtaining of fine-grained structure, comprise namely the following ones [8-18]:

- ☐ High Pressure Torsion
- ☐ Equal Channel Angular Extrusion
- ☐ Cyclic Channel Die Compression
- ☐ Cyclic Extrusion Compression
- ☐ Continuous Extrusion Forming
- ☐ Accumulative Roll Bonding
- ☐ Constrained Groove Pressing

This paper informs namely on ECAP technology investigations that have been oriented by overall objectives of acquiring new knowledge concerning deformation resistances, stress condition impacts, structure and properties of Mg-Zr light metal alloy.

## 2 EXPERIMENTAL PROCEDURES AND RESULTS

The principle of ECAP technology is showed in Fig. 1 a, b. A new tool with basic geometry given in bracket ( $R1 = 4 \text{ mm}$ ,  $R2 = 0.5 \text{ mm}$ ,  $\Psi = 90^\circ$ ,  $\Phi = 90^\circ$  and  $b = 10 \text{ mm}$ ) and channel deflection by  $10^\circ$  (see Fig. 1c.) for experimental procedures was applied at the department of Mechanical Technology [7]. The working site of development of new technologies has at its disposal a hydraulic press of the type DP 1600 kN. For investigation new developed Mg-Zr alloy was used [8]. Chemical composition of the Mg-Zr alloy is given in Tab.1.



**Fig. 1** Principle of ECAP (a, b) and tool with channel deflection by 20° (c)

**Tab. 1** Chemical composition of the Mg-Zr alloy

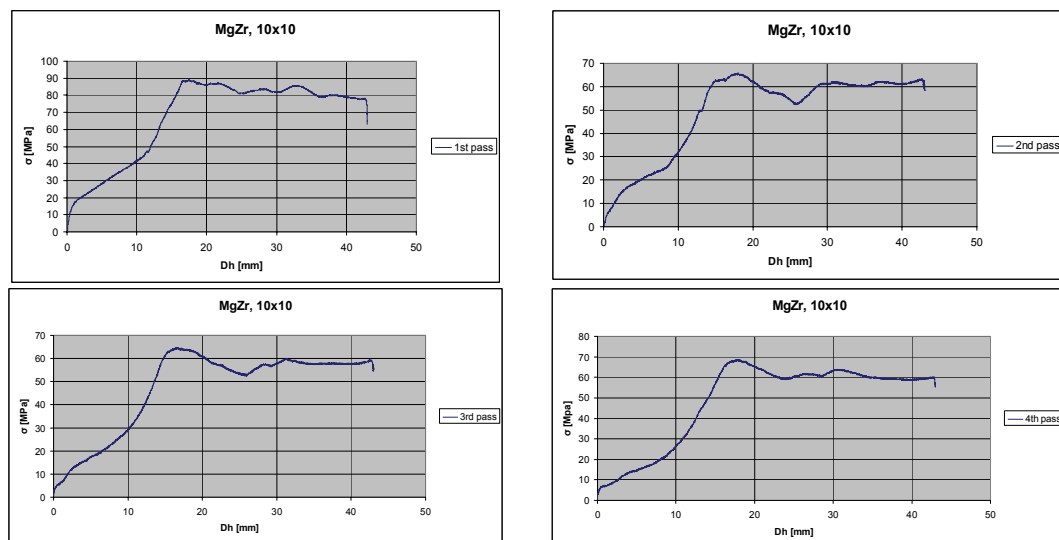
Alloy	Zr	Al	Zn	Mn
Mg-Zr	0,07	0,07	0,01	0,02

## 2.1 Summary of findings at ECAP process

The ECAP forming was realised at condition described above under temperature above 220 °C.

At this temperature the total number of 3 passes was applied in dependence of the development of extrusion [9]. At 4<sup>th</sup> pass at this temperature the sample was injured. From this reason the temperature was increased to 300 °C. Extruded material then was divided into individual series for manufacture of individual testing specimens for metallographic evaluation and mechanical tests in combination the 1<sup>st</sup> - 4<sup>th</sup> pass.

Examples of obtained curves of strengthening in choice passes are shown in the Fig. 2.



**Fig. 2** Examples of obtained curve of strengthening (1<sup>st</sup> - 4<sup>th</sup> pass)

As it is seen from this figure the maximum value of strengthening is reached at the 1<sup>st</sup> pass. At the 2<sup>nd</sup> pass this value decrease and continue to the 4<sup>th</sup> pass approximately on the same level.

## 2.2 Measurement of Vickers hardness and metallographic analyse

The extruded material after all passes was then cut into individual series for manufacture of individual testing specimens for metallographic evaluation and mechanical tests in parallel and up-right direction of deformation.

Mechanical properties by Vickers hardness method were tested. Results of Vickers hardness method are showed in Tab. 2. Average values of hardness in this picture from five measurements were calculated. These values in both cuts slightly increase from the 1<sup>st</sup> to 3<sup>rd</sup> passes. After the 4<sup>th</sup> pass this value is slightly decreased from the reason different condition at experiment procedure (Higher temperature as it is described above).

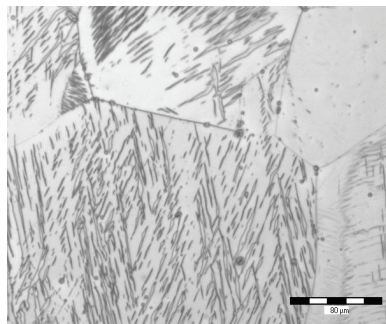
**Tab. 2** Results of Vickers hardness (upright cut)

No of pass	Initial state	1 <sup>st</sup> pass	2 <sup>nd</sup> pass	3 <sup>rd</sup> pass	4 <sup>th</sup> pass
HV5	27	36	35	35	33

The samples for metallographic evaluation were prepared in usual manner. Polishing of samples was made in two stages. In the first stage the samples were polished on cloth with use of the Al<sub>2</sub>O<sub>3</sub> based polishing suspension. In the second stage the polishing was made on very fine velvet cloth with short fibres. Diamond powder with grain size of 1 µm was used as polishing material. Diamond was applied by spraying and cloth was regularly wetted by alcohol-based liquid. The samples were finally flushed with water and spirit and dried by stream of hot air. The samples were then etched by Nital. Duration of etching varied from 5 to 10 seconds.

Light microscope NEOPHOT 2 was used for evaluation of microstructure of alloys.

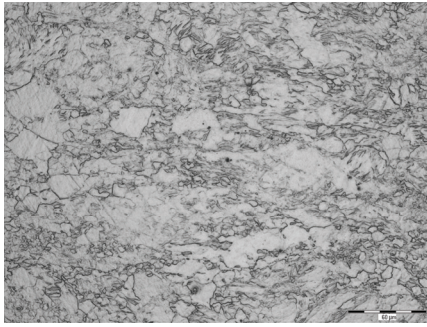
Microstructure of the alloy Mg-Zr in initial state is shown in Fig. 3. Microstructure of the alloy with zirconium is formed mostly by equi-axed grains of various sizes, which contain oblong particles. Due to the fact that the alloy with this composition has been developed recently and its structure is not described in available literature, it can be assumed that these can be grains of magnesium based solid solution, in which a precipitation of fine minority phases could have occurred during solidification due to influence of positive solubility coefficient [10]. However, due to orientation of these etch patterns it is impossible to completely exclude the possibility that these are so called artefacts caused by imperfect removal of the deformed surface layer on the cut. It is also possible to take into consideration forming of a relief at surface deformation during preparation of the sample, or decoration of possible twins or glide bands enriched by dissolved zirconium.



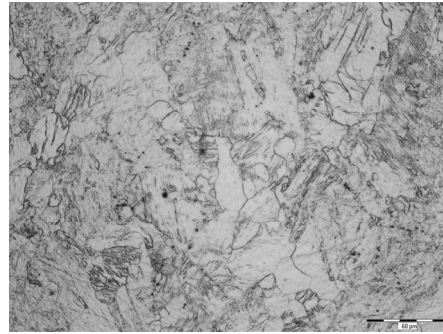
**Fig. 3** Microstructure of the alloy Mg-Zr in initial state

Microstructure of the alloy Mg-Zr after applied deformation (1<sup>st</sup> - 4<sup>th</sup> pass) is shown in Fig. 4. As it is seen from this figure fine grain microstructure was occurred after deformation. The most change of microstructure after the 1<sup>st</sup> pass is detected while after 2<sup>nd</sup> and 3<sup>rd</sup> pass refinement shows lower value. After the 4<sup>th</sup> pass a little increasing of grain is occurred from the reason different condition at experiment procedure (Higher temperature as it is described above).





the 1<sup>st</sup> pass (parallel cut)



the 1<sup>st</sup> pass (upright cut)



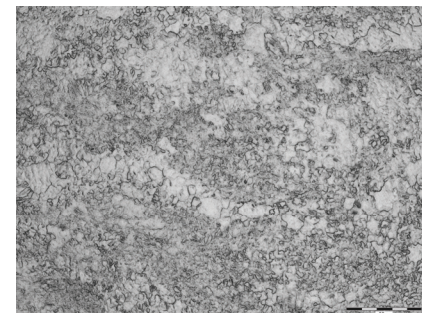
the 2<sup>nd</sup> pass (parallel cut)



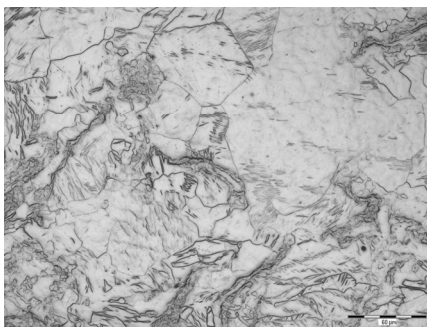
the 2<sup>nd</sup> pass (upright cut)



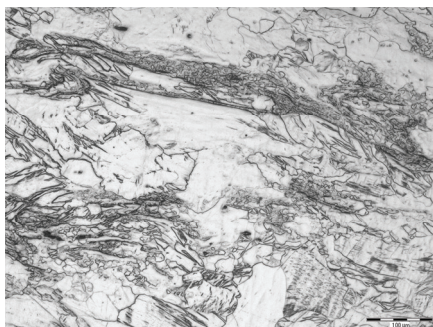
the 3<sup>rd</sup> pass (upright cut)



the 3<sup>rd</sup> pass (upright cut)



the 4<sup>th</sup> pass (parallel cut)



4<sup>th</sup> pass (upright cut)

**Fig. 3** Microstructure of the alloy Mg-Zr after forming

### 3 CONCLUSIONS

On the basis of obtained results it is possible to draw the following conclusions:

- ❑ The ECAP process on Mg-Zr alloy was the first time applied on new developed die and the bottom channel of the tool was deflected by 10° and slightly expanded in its output part.
- ❑ Microstructure of initial state of the Mg-Zr alloy is formed by large polyedric grains of Mg based solid solution with dimensions in the range of 100 – 500 µm.
- ❑ The influence of the changed design of the ECAP tool was unequivocally confirmed.
- ❑ Maximum value of strengthening is reached at the 1<sup>st</sup> pass. At the 2<sup>nd</sup> pass this value decrease and continue to the 4<sup>th</sup> pass approximately on the same level.
- ❑ Metallographic evaluations microstructures of the Mg-Zr alloy have also confirmed more intensive refining of grains already after the 1<sup>st</sup> pass.
- ❑ For reaching the better results on Mg-Zr alloy more experiments at variable conditions will be applied.

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